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SOLVENT EXTRACTION OF PCB CONTAMINATED SOILS BENCH AND PILOT SCALE TESTS

**USEPA REGION III
MINDEN, WEST VIRGINIA
PROJECT 2364-E52**

SUBMITTED TO:

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On-scene Coordinator**

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Findlay, Ohio**

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DRAFT COPY NO. 1

June 25, 1985

Mr. Robert E. Caron
United States Environmental
Protection Agency, Region III
841 Chestnut Street
Philadelphia, PA 19107

Dear Bob:

Here is a draft copy of our final report for the extraction of PCBs from soils of the Minden, West Virginia, site. There are several important considerations I would like to share with you before you read the report.

First, this is a draft, and you are the only one who has seen this outside of OHM. I anticipate that after your review, we can jointly change or modify portions to better suit its final audience and then send out a copy for general distribution at EPA.

Second, the theme of the report is primarily technical feasibility and secondly economic feasibility. I thought that in the best interest of introducing new technology, it should be evaluated financially as generic unit processes (i.e., based on published costs/unit prices without influence by any one contractor or another). Let me know your thoughts on this.

Third, the cost evaluation does not represent our bid for performing the work. I would like the opportunity to provide a stand-alone proposal at the appropriate time to describe our technical approach and costs. Obviously, we are interested in pursuing the project, and the bid costs would be structured more closely to current industry formats for unit prices as with the ERC's Catalog Price List.

Mr. Robert E. Caron

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June 25^{OR} 1985
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I plan to be in Florida for two weeks, but I will be in direct contact with our corporate office every day and will call you with numbers to reach me once the job site is set up. I look forward to working with you on this project.

Sincerely,

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Manager, Projects
Research and Development

SI:dmm

Enclosure

pc: Project File No. 2476

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I. EXECUTIVE SUMMARY

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The cleanup of hazardous and industrial sites has recently been hampered due to limitations in the disposal of large volumes of contaminated soils and/or sediments. To date, conventional disposal of these materials has been accomplished in secured hazardous waste landfills. However, recent scientific studies have initiated concerns over the containment integrity of these landfills. These concerns are based on the wastes' longterm ability to alter the containment properties and possibly enhance leaching of the contaminant to the surrounding soils and ground water. This has caused a general trend in industry to seek alternate, more complete methods of treating wastestreams previously designated for landfilling.

One such method under consideration is to apply continuous, countercurrent extraction systems to the problem of hazardous waste reduction.

The technology of extraction has been used extensively for testing and analyses but has not been formally evaluated for large-scale removal of contaminants from soil.

The purpose of this study is to define and evaluate the unit processes necessary to promote the extraction of polychlorinated biphenyls (PCBs) from contaminated soil currently stockpiled at the Shauffer Equipment Company (SHAUFFER) site in Minden, West Virginia. The primary focus of these investigations was to enhance material handling techniques in order to promote continuous and consistent flows through the equipment applicable to a full-scale process treating a maximum of 10 tons per hour (TPH) of soil.

The technical approach consisted of two testing phases: 1) benchscale extraction tests with an initial characterization of soil and various physical and chemical parameters; and 2) pilot field investigations to evaluate solvent selections and optimize operational requirements. All testing was performed on site using a mobile laboratory and pilot equipment sized to evaluate low flow rates of contaminated soil.

Following the completion of both programs, the results indicated that a process consisting of a materials handling system and a countercurrent extractor is effective in removing PCBs from the contaminated soil into the contacting solvent. The use of both methanol and methanol/FREON TF mixtures demonstrated removal efficiencies approaching 90 percent for single extractions, with 90 to 95 percent reductions possible for multiple-pass extractions. FREON TF alone did not appear feasible under certain test conditions due to physical interactions with the soil.

These tests clearly demonstrated that a 10 TPH, full-scale extraction system to remove PCBs is technically feasible based upon parameters optimized during the benchscale and pilot program. A host of unit processes are available to mitigate the contamination at SHAUFFER and it should be noted that these are site-specific in that others may be more feasible elsewhere.

II. INTRODUCTION

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The following test program was developed to define the unit processes necessary to promote the extraction of PCBs from contaminated soil at SHAUFFER in Minden, West Virginia.

The technical approach was comprised of two testing phases: (a) benchscale modeling of extraction procedures with a characterization of physical and chemical parameters, and (b) pilot trials to evaluate and optimize the extraction volumetric ratios and contact times.

III. TECHNICAL APPROACH - OHM EXTRACTION STUDIES ^{ORIGINAL} (Red)BENCHSCALE TESTING PROGRAM

Benchscale testing was organized into three categories:

- o Substrate Sizing and Classification
- o Substrate Chemical/Physical Characterization
- o Substrate/Solvent Interactions at Various Ratios

All of the above benchscale tests were performed on site using a mobile laboratory and contaminated soil from the clay-lined storage area.

Substrate Sizing and Classification

Substrate sizing and classification tests were performed under the guidelines listed in the ASTM Method D422 (04.08), "Particle Size Analysis of Soils." This method defines soil particle size by passing the soil through a series of particle sieves and determining the distribution based on either the number of particles or their mass equivalents retained or passed by that particular sieve. Sieve sizes ranging from 2 inches to a No. 100 mesh were originally proposed for classification testing. During the actual testing program, however, it became necessary to redefine the selection of sieves following initial collection of the soil. With the likelihood of particle buoyancy in tested solvents, soil sizing and classification was necessary in order to optimize pilot scale equipment and solvent flow rates.

The first benchscale test was used to define gross interactions of the substrates with the solvents proposed for use (i.e., observations for particle settling, dispersions, buoyancy, and emulsions). Contaminated soil and control soil samples were added to a predetermined volume of each test solvent. Eight graduated cylinders were used for the test (four contaminated, four control). 100 ml of solvent was added to each cylinder, followed by a measured amount of soil. The cylinders were stoppered, shaken for 10 seconds, and allowed to settle undisturbed. Observations were made for each cylinder to check for settled particles, particle dispersions, buoyant particles, and emulsions. After one minute, the solvent was slowly decanted through a tared filter paper taking care to remove only suspended and buoyant particles. The filters were allowed to air dry and reweighed to determine the gross amount of fines encountered. The soil residues remaining at the bottom of the cylinders were rinsed over to 500 ml beakers, decanted and air dried as before.

Two solvents were initially used for this test: FREON TF and a 50/50 mixture of FREON TF/methanol. The following table identifies the test samples. ^{ORIGINAL}_(Red)

TABLE 1

<u>Sample</u>	<u>Description</u>
A	Contaminated soil/FREON TF wash
B	Contaminated soil/FREON TF wash
C	Contaminated soil/FREON TF/ methanol
D	Contaminated soil/FREON TF/ methanol
E	Control soil/FREON TF wash
F	Control soil/FREON TF wash
G	Control soil/FREON TF/methanol wash
H	Control soil/FREON TF/methanol wash

Further experiments were carried out using variable ratio FREON/methanol mixtures to observe the soil/solvent interactions. Approximately 50 grams of contaminated soil was placed in an 8-ounce sample jar. A total of 100 ml of the selected solvent was added to the sample followed by brief swirling of the jar contents. After allowing the samples to settle undisturbed, observations were made of the soil/solvent interactions. The solvent mixtures used were as follows:

TABLE 2

<u>Sample</u>	<u>Description</u>
A	100 percent FREON TF
B	90 percent FREON TF/10 percent methanol
C	80 percent FREON TF/20 percent methanol
D	70 percent FREON TF/30 percent methanol

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<u>Sample</u>	<u>Description</u>
E	60 percent FREON TF/40 percent methanol
F	55 percent FREON TF/45 percent methanol
G	50 percent FREON TF/50 percent methanol
H	45 percent FREON TF/55 percent methanol
I	40 percent FREON TF/60 percent methanol
J	30 percent FREON TF/70 percent methanol
K	20 percent FREON TF/80 percent methanol
L	10 percent FREON TF/90 percent methanol
M	100 percent methanol

A third test was performed utilizing FREON TF/methanol mixtures with sized and unsized soil to determine whether a classification of the material would affect the soil/solvent interactions observed earlier. This was done using control samples only.

TABLE 3

<u>Sample</u>	<u>Description</u>
A	FREON TF added to unsized soil
B	FREON TF added to soil < 2 mm in size
C	50 percent FREON TF/50 percent methanol added to unsized soil
D	50 percent FREON TF/50 percent menthanol added to soil < 2 mm in size

Each sample jar was swirled, the contents were allowed to settle, and were then observed.

It soon became obvious that the polar solvent (i.e., methanol) formulations dispersed the large soil agglomerates into smaller fines after treatment. This test was designed

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to observe the dispersion of soil particles into fines as a result of the treatment, by sizing the contaminated soil before and after contact with selected solvents. Four contaminated soil samples were weighed and placed in glass beakers. Two of the samples were swirled with methanol for ten minutes; the solvent was then decanted from the soil and the sample was allowed to air dry. All four samples were then sized with the shaker using the various screens.

TABLE 4

<u>Sample</u>	<u>Description</u>
A	Control - no wash
B	Control - no wash
C	Methanol wash
D	Methanol wash

Further experiments were conducted to determine the distribution of PCBs according to soil particle size. An aliquot of the contaminated soil was classified into the different size fractions listed below:

TABLE 5

<u>Sample</u>	<u>Description</u>
A	Soil > 6.30 mm
B	Soil < 6.30 mm and > 2.00 mm
C	Soil < 2.00 mm and > 0.425 mm

Each size fraction was then analyzed for PCB content.

Substrate Chemical/Physical Characterization

The chemical/physical characterization of the substrate was required to optimize solvent/substrate contact during the continuous extraction process. The basis of this testing was focused around several perceived problems:

- o Interference of the extraction solvent with excess water to form emulsions
- o Non-uniform water dispersion in the discrete soil particles to hamper penetration by the solvent and reduce extraction efficiencies
- o Heterogeneous organic distribution in the bulk staged soils which, when processed, may temporarily overload the extraction system.

Samples were tested for moisture content, organic content, and inorganic content using the gravimetric residue analysis as outlined in "Standard Methods for the Examination of Water and Wastewater," (Method 209A Total Residue

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Dried at 103-105 degrees Celsius, Method 209 E Total Volatile and Fixed Residue at 550 degrees Fahrenheit).

Ten soil samples, comprised of five control samples and five contaminated samples, were sieved so as not to contain any fractions greater than 2 mm in size. Each sample was initially weighed and then dried at 105 degrees Celsius to drive off moisture. The soils were reweighed so that a determination of total percent solids could be made. Next, two samples of the dried control and contaminated soils were heated to 550 degrees Fahrenheit as detailed in Method 209 E to calculate the organic and inorganic content of the two soil types.

The next test was designed to examine any possible interferences to extraction from the presence of excess water in the medium. This test was initiated by first collecting a total of six samples, comprised of three control samples and three contaminated samples. These soils were sized so as not to contain fractions greater than 2 mm in size. The samples were activated at 550 degrees Fahrenheit for 1 hour, followed by dousing with water at varying weight percents. These mixtures were added to 100 ml graduated cylinders and 50 ml of FREON TF was poured in. The cylinders were stoppered and shaken for 10 seconds. Each sample was observed for dispersions, soil clumping, and emulsions.

Additional tests were performed to calculate the bulk density of various soil types used in the pilot extraction trials. This test was designed to furnish information pertinent to the soil addition rates so that a more accurate representation of PCB mass transfer could be achieved. In general, each of the soils tested in this experiment was typical of influent solids extracted with the pilot contacting unit or washed solids leaving this.

For these density determinations, a particular sample of the contaminated soil was added to a prepared 40 ml glass beaker and tapped lightly for a few seconds. Incremental amounts of soil were continuously added until the apparent volume of soil was 40 ml. The procedure was repeated four more times for each soil type. After recording the weight in grams the three most consistent values were averaged to calculate the bulk density. Table 6 lists the various soil types that had been prepared for the field extraction trials.

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TABLE 6

<u>Sample</u>	<u>Description</u>
17	Wet soil passed through a 2 mm sieve, crushed, and dried extremely well
34	Washed soil initially less than 2 mm, dried extremely well, and contacted with FREON TF
35	Wet soil passed through a 2 mm sieve, and dried extremely well
37	Washed soil initially added as less than 10 mm, dried extremely well, and contacted with FREON TF
38	Pile composite wet soil passed through a 2 mm sieve, and then dried
69	Wet soil passed through a 10 mm sieve
76	Washed soil initially added as less than 10 mm wet, contacted with the 50 percent FREON TF/50 percent methanol mixture, and dried
80	Wet soil passed through a 10 mm sieve, then dried
82	Washed soil initially added as less than 10 mm, dried extremely well, and contacted with the 50 percent FREON TF/50 percent methanol mixture
95	Wet soil passed through a 10 mm sieve

Substrate/Solvent Interactions at Various Ratios

The purpose of these tests was to evaluate the extraction of PCBs as a function of solvent-to-soil mass ratios using selected solvents. This, in turn, indicated the economic feasibility of using that particular solvent and the removal efficiency which could be anticipated. Similarly, the tests were also useful in predicting the highest solubility of PCBs (solvent capacity) which could be achieved by the solvent. This information is important in the sizing of pilot scale and full scale equipment to accommodate the expected flow rates once the extraction process has been optimized.

Three specific solvents were selected for this experiment, FREON TF, methanol, and a 50/50 mixture of the two. In each case, five contaminated soil samples were each weighed to 50 grams and placed in separate mixing jars. Different volumes of each solvent were added to each jar so that a range of solvent-to-soil ratios is encountered for the extraction. Solvent-to-soil mass ratios ranged from 1.14:1 to 10.9:1 for methanol, 2:1 to 20:1 for FREON TF, and 2.4:1 to 11.8:1 for the 50/50 mixture. The soils were extracted by mixing with the solvent for 30 minutes using a magnetic stirrer. After mixing, the solvent was decanted from the soil and analyzed for PCBs. The extracted soil was reextracted with the analytical method to determine the PCB content remaining in the soil.

Extraction Efficiency Versus Temperature of the Solvent

Tests were also conducted to determine whether heated solvent in contact with contaminated soil could improve extraction efficiencies over the same solvent at ambient temperature. If significant increases were recorded, substantial savings in equipment size and costs might be possible.

Four contaminated soil samples were weighed to approximately 50 grams. All samples were then extracted with 320 ml of FREON TF at temperatures ranging from 73 degrees Fahrenheit up to 115 degrees Fahrenheit near the boiling point of this solvent. The samples were mixed for 30 minutes with a magnetic stirrer while maintaining the respective temperature. The solvent was then decanted and analyzed for PCB content. The extracted soil was analyzed by sonication to determine the remaining PCB content.

PILOT SCALE FIELD INVESTIGATIONS

Following the completion of preliminary benchscale testing, a pilot scale field investigation was performed. The purpose of these extraction tests was to determine the most effective solvent for the removal of PCBs from the stockpiled soil at the site. In addition, repetitive trials were run with the best solvent to establish potential removal efficiencies for a scaled up system. The choice of solvent should also be dependent upon anticipated cost savings over other solvents, and should exhibit low toxicity, low volatility, easy containment, and considerably easy recovery for reuse while on site.

Introduction

Operational Parameters

A continuous countercurrent contactor was chosen as a pilot treatment method for chemical extraction of the PCBs. In a continuous countercurrent extraction, the flow of the

solvent is opposite to that of the soil so that the contaminated soil contacts a liquid of increasing purity. Specifically, several operational parameters directly affect the performance and extractive efficiency of the contactor:

- o Type of soil encountered
- o Solvent used
- o Particle size, shape, and moisture content
- o Solvent-to-soil mass ratio
- o Influent PCB concentration
- o Residence time of the solids contacting the solvent
- o Degree of mixing between soil and the solvent

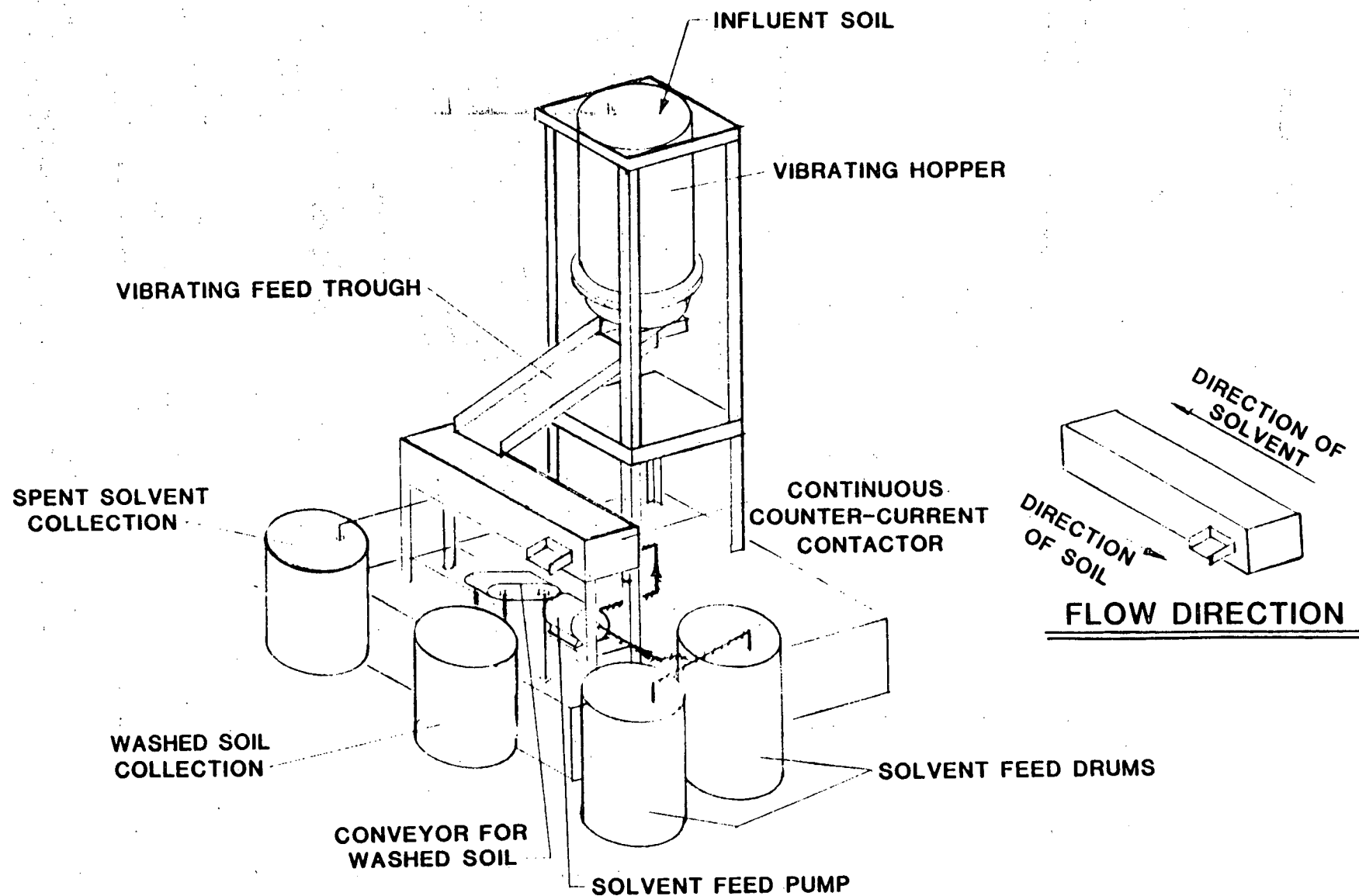
These other parameters were varied during the course of the pilot extraction experiment in an attempt to define the optimum conditions under which the highest removal efficiency for PCBs could be anticipated.

Pilot Treatment Equipment

The equipment comprising the pilot treatment system included a vibrating hopper and feed trough for soil addition, a GREERCO stainless steel contactor, solvent feed system including a 0.75 Hp Oberdorfer plastic gear pump, Fisher and Porter rotameter, and influent piping. The washed soil was conveyed to pails or holding cells.

Contactor Operating Principle

Contaminated soil was manually bucketed into the top of the feed hopper. Once the desired feed rate was established, both the hopper and feed trough controls were preset to maintain a relatively constant flow rate and consistency. Soil then entered the contactor through a port located on the side. The contactor itself was horizontal with only a slight incline to allow the solvent to flow by gravity over the contaminated soil. The conveying means consisted of a stainless steel shaft at the center of the trough to which were fastened adjustable paddles. The paddles formed an uninterrupted helix to propel the solids forward and yet offer little resistance to the counter flow of solvent. Maximum contact with the liquid was achieved by overlapping the paddles and creating a tumbling action through the trough. At the end of the trough, a collection plate transferred the washed solids out of the contactor and onto a belt conveyor (See Figure 1).



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SCHEMATIC OF PCB PILOT EXTRACTOR

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The solvent used for the extraction testing was supplied in drums to the site. Each of these drums was situated next to the equipment structure so that the influent feed pipe and foot valve could be quickly coupled for easy access. An Oberdorfer plastic gear pump then fed the solvent through a Fisher and Porter rotameter for flow measuring. The scale on the flowmeter was given as a percentage of full scale flow, and therefore it became necessary to calibrate the scale to measure FREON TF, which is significantly denser than water at ambient temperatures. Extremely low flow rates could be achieved through the installation of a bypass circuit to divert fresh FREON TF back to the feed drum. This flow was regulated by either a plug valve located on the meter itself or a ball valve situated on the bypass line. Solvent leaving the flowmeter was piped into the contactor unit using $\frac{1}{2}$ -inch carbon steel line. A variable speed, explosion-proof motor controlled the paddle speed and hence the residence time of the contaminated soil in the extractor. A process diagram is given in Figure 2.

Pilot Extraction Program: (Test No. 5)

Introduction

Pilot extraction tests were performed on site using the GREERCO countercurrent contactor and related equipment illustrated in Figure 1. This particular solids-liquid separation unit measured 4 feet in length and 5 inches across; maximum residence time in the contactor could approach 2 hours if necessary. Contaminated soil and the chosen solvent were introduced at rates necessary to duplicate the solvent-to-soil ratios utilized in the benchscale tests with that solvent. These solvent-to-soil ratios yielded distinct PCB removal efficiencies (see section entitled "Results of the Solvent-to-Soil Ratio Testing.")

In order to create a large data base from which to draw conclusions concerning the extraction of PCBs from the soil, OHM devised a pilot treatment program which investigated many of the variables that could affect extractive efficiency. A list of these variations is given below:

- o Solvents Used
 - Methanol
 - FREON TF
 - 50 percent FREON TF/50 percent methanol
 - 75 percent methanol/25 percent FREON TF
- o Particle Size of the Soil
 - less than 2 mm
 - less than 10 mm
 - Finely ground powder

VIBRATING HOPPER

VIBRATING FEEDER

FI

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CONTACTOR

VARIABLE SPEED

WASHED SOLIDS

CONVEYOR

SPENT SOLVENT

FRESH SOLVENT

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PROCESS DIAGRAM

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whether the extractive power of the solvent had been diminished at all, and to what extent the PCBs could be further solubilized into the solvent.

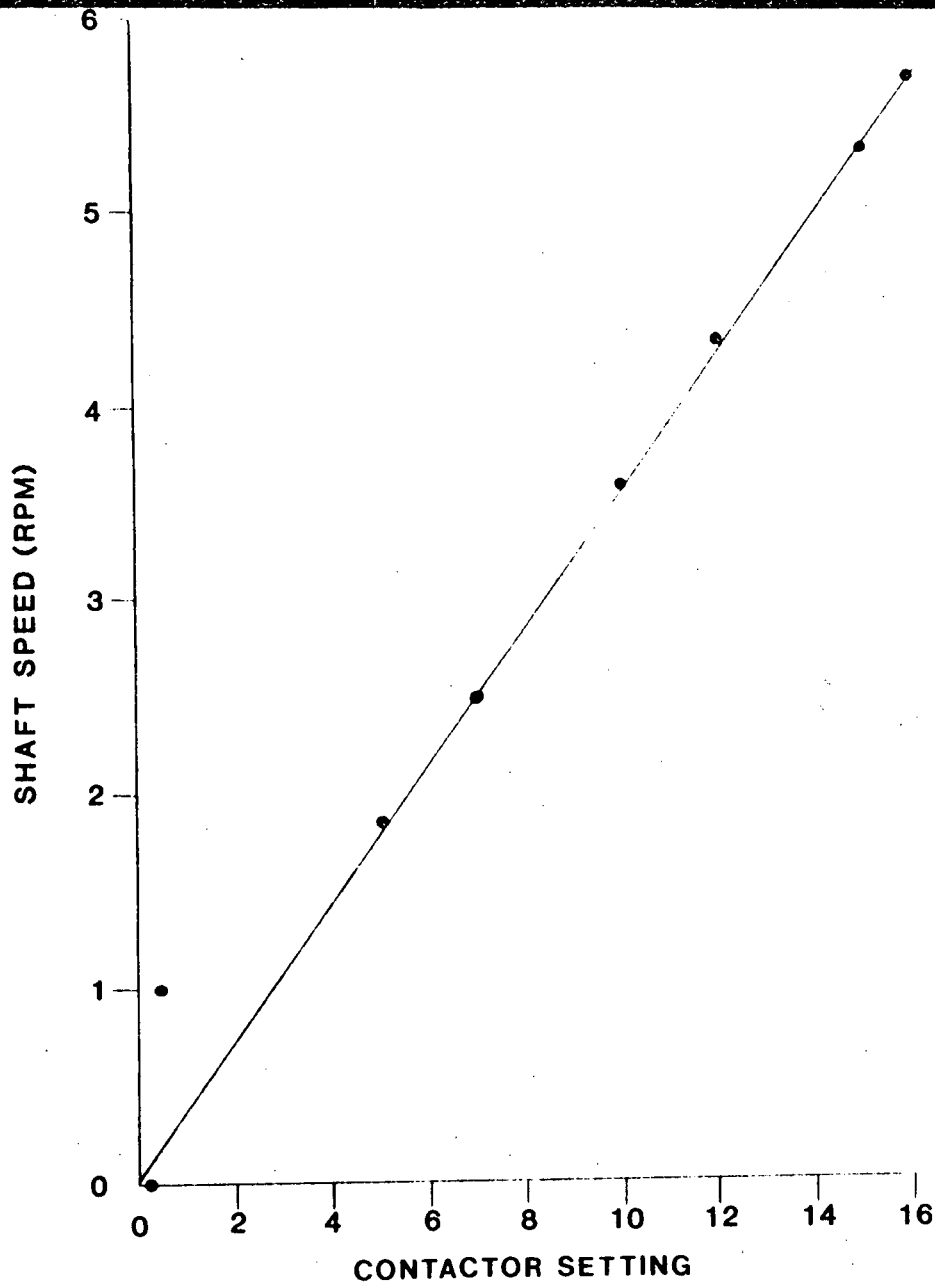
Testing Procedures

Before implementing a trial run, all operating parameters had to be defined so that hopper, trough, extractor, and pump settings could be established. For each run, a decision was made as to:

- o Particle size of the soil to be tested
- o Wetness of the influent soil (wet, dry, or extremely dry)
- o Influent contaminated soil feed rate
- o Choice of solvent used
- o Flow rate of the solvent
- o Residence time of the soil in the contactor

To begin a trial run, the contactor and related soil handling system was thoroughly decontaminated with methanol and various cleaning agents to remove any traces of soil particles. A final rinse of FREON TF removed any traces of moisture and dried the contacting system. Once the residence time was chosen, the contactor shaft speed in revolutions per minute was derived using correlations available from the GREERCO contactor literature. Shaft speed was then transposed over to a corresponding contactor setting (Figure 3). The solvent was added to the contactor trough until the liquid level covered half of the drive shaft. This required about 2 gallons of solvent. Next the contactor drive was started and both soil and liquid feed systems were initiated. Samples of the influent soil were taken, in addition to washed soil samples after the solids had begun to exit the contactor at the opposite end. Similarly a sample of the used solvent was collected for analysis. After samples were taken at the various stations the contactor speed setting was increased to expedite the removal of the remaining solids onto the conveyor. The solvent contained in the contactor trough was drained through a port on the underside. Decontamination procedures were then initiated to prepare for another trial.

The first phase of the pilot extraction tests consisted of trials conducted with pure solvents and variations of the soil conditions. Both wet and dry materials were examined at 2 and 10 mm sizes while using a range of solvent-to-soil ratios. For each classification of soil type (i.e., dry and



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O.H.M.

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less than 10 mm) several residence times were investigated if possible. Holding all other parameters constant would then demonstrate the effect of residence time on PCB extractive efficiency. Both FREON TF and methanol were the solvents selected for this phase of the program. (See Figure 4.)

Preliminary results from these initial trials suggested that a combination of solvents might be practical and quite efficient in extracting PCBs from the contaminated soil. The second phase of testing investigated various combinations of FREON TF and methanol:

- o 50 percent FREON TF/50 percent methanol
- o 75 percent methanol/25 percent FREON TF

Furthermore, only two variations of the soil were used for the feed solids.

- o Soil less than 10 mm and dried extremely well
- o Soil less than 10 mm and taken directly from the holding cell

This particle size appeared to represent a fraction which could be handled well by the full scale process. Once again, several trials were performed for each soil type and solvent-to-soil ratio to investigate the effect of residence time.

The third phase of testing utilized multiple passes through the contactor in order to further reduce residual PCB levels. In the first set, wet soil screened less than 10 mm in size was added to the contactor as before, except now in the absence of solvent in the trough. Methanol was sprayed over the tumbling soil as it was carried over to successive paddles. Spent solvent from the contactor was collected and analyzed for PCB content. The washed soil was conveyed into a clean pail after a contactor residence time of 5 minutes. A sample of this soil was similarly taken for analysis. After decontaminating the extraction system, the collected and washed soil was reintroduced into the extractor, this time utilizing pure FREON TF as the extractive solvent. The soil residence time was set for 15 minutes. The washed soil from this trial was likewise collected and a small fraction of this analyzed for PCBs. Following decontamination of the unit a third pass was accomplished with pure FREON TF. Solvent and washed soils were tested for residual PCBs.

This same procedure was repeated for wet soil less than 10 mm in size, yet this time using a 50 percent FREON TF/50 percent methanol mixture as the extractive solvent. Residence times were 30 minutes for each pass with solvent-to-

SOIL CATEGORIES STUDIED

EXTREMELY DRY & < 2MM
 DRY & < 2MM
 WET & < 2MM
 EXTREMELY DRY & < 10MM
 DRY & < 10MM
 WET & < 10MM

SOIL ADDITION RATES STUDIED

3 - 10 lb/HR
 13 & 20 lb/HR
 30 & 320 lb/HR

SOIL PROPERTIES:

% MOISTURE
 DENSITY
 PARTICLE SIZE
 PCB DISTRIBUTION
 ON PARTICLES
 BUOYANCY

SOLIDS FEED

SCREENING TECHNIQUES

SPENT SOLVENT

SPENT SOLVENT STUDIED FOR :

- RESIDUAL PCB ON FINES
- SOLUBILITY OF PCBs IN
- FILTERABILITY OF SOLIDS
- PARTICLE SIZE OF SOLIDS
- EXTRACTIVE CAPABILITY ON 2ND & 3RD PASSES

RETENTION TIMES:

5 MIN
 10 MIN
 15 MIN
 30 MIN
 1 HR

TEMPERATURE EFFECTS

50% MEOH / 50% FREON
 STUDIED

SOLVENT TO SOIL RATIOS:

1-12:1
 80-90:1

SOLVENT UTILIZED:

FREON 113
 METHANOL
 50% MEOH / 50% FREON
 75% MEOH / 25% FREON

SOLVENT ADDITION RATES STUDIED:

0.025 - 0.16 GPM
 0.40 - 0.60 GPM

SOLVENT FEED

COUNTER-CURRENT GREERCO CONTACTOR UNIT

WASHED SOLIDS

SOIL PROPERTIES:

% SOLVENT RETAINED
 % WATER
 DENSITY
 PARTICLE SIZE

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FIGURE 4

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solvent feed rates would not require such an extensive recycling effort than that needed for higher solvent-to-soil ratios. This would also result in smaller chemical feed systems and full-scale extractor volumes. Most importantly, a significant cost-savings would be realized for up-front purchases of pure solvent. Solvent-to-soil ratios used in the pilot study were selected from the results of the benchscale study.

Changing the solvent-to-soil ratio in the field pilot extraction study was accomplished in two ways:

- o Raising or lowering the solvent feed rate while fixing the soil addition rate
- o Increasing or decreasing the soil addition rate while holding the solvent feed rate constant.

Both the solvent and soil flow rates are normally expressed as pounds per hour, so that the solvent-to-soil ratio is therefore a dimensionless term having no units at all.

Residence Time in the Contactor

An equally important variable evaluated during the pilot extraction trials was the residence time of the contaminated soil in the contactor unit. In this instance the residence is defined as the time that the contaminated soil is in contact with the solvent. Pilot trials conducted with the extraction equipment attempted to show how PCB removal efficiency was altered in response to variations in this residence time.

Moisture Content of Influent Soil

Field extraction trials also addressed the moisture content of the contaminated soil and how it affected PCB removal efficiencies as a result of solvent/soil interactions. These interactions were examined in the benchscale studies by controlling the amount of water in the entering soil. For the pilot tests, heated enclosures were constructed to dry the contaminated material. After remaining in the enclosure for approximately one day, the moisture content had been lowered to under 0.5 percent in the soil. During the extraction trials both these wet and dry soils were contacted with the solvent and observations and efficiencies were recorded.

Solvent Purity

Although most of the pilot trials were performed with pure solvent, two sets utilized solvent that had previously contacted contaminated soil. These tests investigated

- o Solvent-to-Soil Ratio
 - 0.82-20:1
 - 85:1
- o Residence Time in the Contactor
 - 3 minutes - 60 minutes
- o Moisture Content of Influent Soil
 - Extremely dry (0.5 percent) up to wet (20 percent)
- o Solvent Purity
 - Pure (100 percent) and solvents containing trace amounts of PCBs from prior trials

Description of Operational Parameters

Solvents Used

A variety of solvents and solvent combinations were introduced into the contactor with the contaminated soil to test for extractive capabilities. Furthermore, each of the solvents selected interacted differently with the contaminated soil and this would likely influence the rate of PCB extraction. Methanol appeared capable of dispersing the soil agglomerates into fine particles, while the high specific gravity of FREON TF initiated buoyancy among particular soil fractions. From these observations it became necessary to field test the solvents in order to investigate their impact upon removal efficiencies. Combinations of the two selected solvents were also investigated to learn whether the useful properties of each could be utilized.

Particle Size of the Soil

Pilot extraction tests were conducted on various size fractions of the contaminated soil in order to investigate the effect upon PCB removal efficiency and the implications these size fractions imposed on the design of a full-scale extraction system. OHM used soil particles a) less than 2 mm in size, and b) 2 to 10 mm in size. These fractions were obtained by screening a sufficient quantity of soil for the trial run through the test sieves utilized during the benchscale program.

Solvent-to-Soil Ratios

The testing of different solvent-to-soil ratios constituted a major portion of the pilot extraction program. From a materials handling viewpoint, it was apparent that lower

soil ratios kept under 5:1. In each run, a pure solvent mixture was used with samples of each stream analyzed for PCB content.

A third variation of the above steps examined the effects of reintroducing used solvent as the extractive liquid. During the first pass, both soil and spent solvent were collected. Washed soil was kept in a closed pail to minimize evaporative losses, while the spent 50/50 mixture was sealed in 1-gallon bottles. For the second pass, the bottled solvent was introduced using an 1800 ml separatory funnel situated at the liquid inlet port. Washed soil and the used solvent were saved a second time for use in the third pass. Samples were taken from each entering and exiting stream during the entire procedure. Residence times remained at 30 minutes.

A fourth phase of Test No. 5 was included to verify the results obtained in the previous trials where fresh solvent and used solvent were compared in multiple passes for extractive efficiency. These two runs were repeated using nearly identical operating parameters as those of the third variation.

The final phase of the pilot extraction testing was a second replicate of the three-pass multiple extraction with fresh solvent added for each successive extraction. Operating parameters remained nearly unchanged from before.

RESULTS

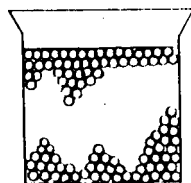
Benchscale Testing Program

Test No. 1

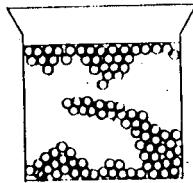
Test No. 1 involved the addition of weighed amounts of solid to investigate settled particles, particle dispersions, buoyant particles, and emulsions.

TABLE 7

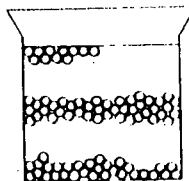
<u>Sample</u> <u>Buoyant</u>	<u>Description</u>	<u>Sample Wt.</u>	<u>Buoy.Wt.</u>	<u>Percent</u>
A	Contaminated soil/ FREON TF wash	27.9759 g	6.6025 g	23
B	Contaminated soil/ FREON TF wash	34.6164 g	9.0946 g	26
C	Contaminated soil/ FREON TF/methanol	32.1236 g	4.7578 g	15



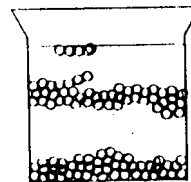
#1
100% FREON



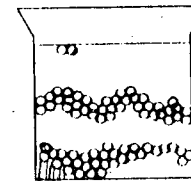
#2
90% FREON/
10% METHANOL



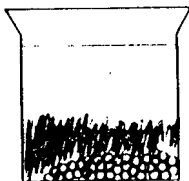
#3
80% FREON/
20% METHANOL



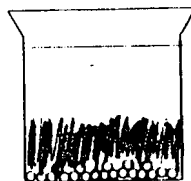
#4
70% FREON/
30% METHANOL



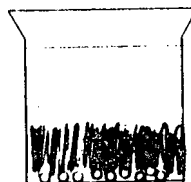
#5
60% FREON/
40% METHANOL



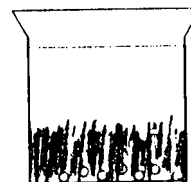
#6
55% FREON/
45% METHANOL



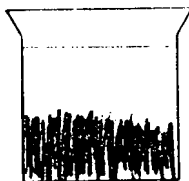
#7
50% FREON/
50% METHANOL



#8
55% METHANOL/
45% FREON



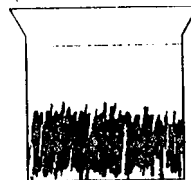
#9
60% METHANOL/
40% FREON



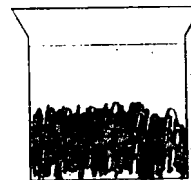
#10
70% METHANOL/
30% FREON



#11
80% METHANOL/
20% FREON



#12
90% METHANOL/
10% FREON



#13
100% METHANOL

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PHYSICAL INTERACTIONS BETWEEN
PARTICLES AND SOLVENT

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FIGURE 5

ORIGINAL
(Red)

<u>Sample No.</u>	<u>Type Soil</u>	<u>Wet Weight, g</u>	<u>Dry Weight, g</u>	<u>ORIGINAL (Red) Percent Solids</u>
54	Contaminated	12.3761	10.4222	84.2
55	Contaminated	11.6259	9.7873	84.2
56	Control	13.0500	10.4905	80.4
57	Control	15.3903	12.3443	80.2
58	Control	12.6855	9.0797	71.6
59	Control	11.0820	8.9300	80.6
60	Control	13.8415	11.1700	80.7

Next, two samples of the dried control and contaminated soils were subjected to heat in order to determine the ash content.

TABLE 12

<u>Sample No.</u>	<u>Dry Weight, g</u>	<u>Ashed Weight, g</u>	<u>Percent Ash</u>
53	10.0146	5.1893	51.8
55	9.7873	5.3017	54.2
57	12.3443	10.3878	84.2
58	9.0797	7.6356	84.1

Both contaminated and control samples showed moisture contents of 15 percent to 20 percent. Upon ashing it was demonstrated that the contaminated samples had a greater percentage of organic material that was volatilized after heating to 550 degrees Fahrenheit.

Test No. 4

Test No. 4 involved the addition of water to ashed contaminated and noncontaminated soil samples to investigate the various interactions between the substrate and solvent.

TABLE 13

<u>Sample</u>	<u>Type Soil</u>	<u>Weight, g</u>	<u>ml H₂O Added</u>	<u>Percent H₂O</u>
1	Contaminated	3.1323	0.75	25
2	Contaminated	2.9239	1.50	50
3	Contaminated	3.0905	3.00	100

<u>Sample</u>	<u>Type Soil</u>	<u>Weight, g</u>	<u>ml H₂O Added</u>	<u>Percent H₂O</u>
4	Control	3.5876	0.85	25
5	Control	3.6113	1.80	50
6	Control	3.5019	3.50	100

After shaking the cylinders, the soil was observed to coat the sides of the glass. Large clumps of soil aggregated at the bottom of the cylinders and some floated to the surface. Control samples and contaminated samples were observed to behave identically in all six cases. The only observed difference was that the 100 percent water-doused samples formed a separate liquid phase on top of the FREON TF. This layer was observed to have dissolved and suspended soil particles.

Density Experiments

Representative samples from each of the various soils encountered during the pilot extraction testing were investigated to determine the bulk density of each.

TABLE 14

<u>Sample No.</u>	<u>Density, g/cc</u>
17	0.66
34	1.22
35	0.92
37	0.98
38	0.94
69	0.87
76	1.13
80	1.07
82	0.97
95	0.87

Solvent-to-Soil Ratio Testing

Laboratory jar tests were conducted in order to observe the PCB removal efficiency as the solvent-to-soil ratios were increased. Three solvents were selected:

- o Methanol
- o FREON TF
- o A mixture of 50 percent FREON TF and 50 percent methanol

These results were useful in predicting the extractive capability of each solvent along with attainable PCB removal efficiencies. The distribution coefficient, a calculated

value obtained through these tests, became important for a normalization of the equilibrium data presented here (see Figures 6, 7, 8, 9).

TABLE 15

EXTRACTION EFFICIENCY OF PCBs VERSUS
SOLVENT/SOIL RATIOS
SOLVENT = 100 PERCENT METHANOL

PCB Concentration in entering soil = 350 micrograms/gram

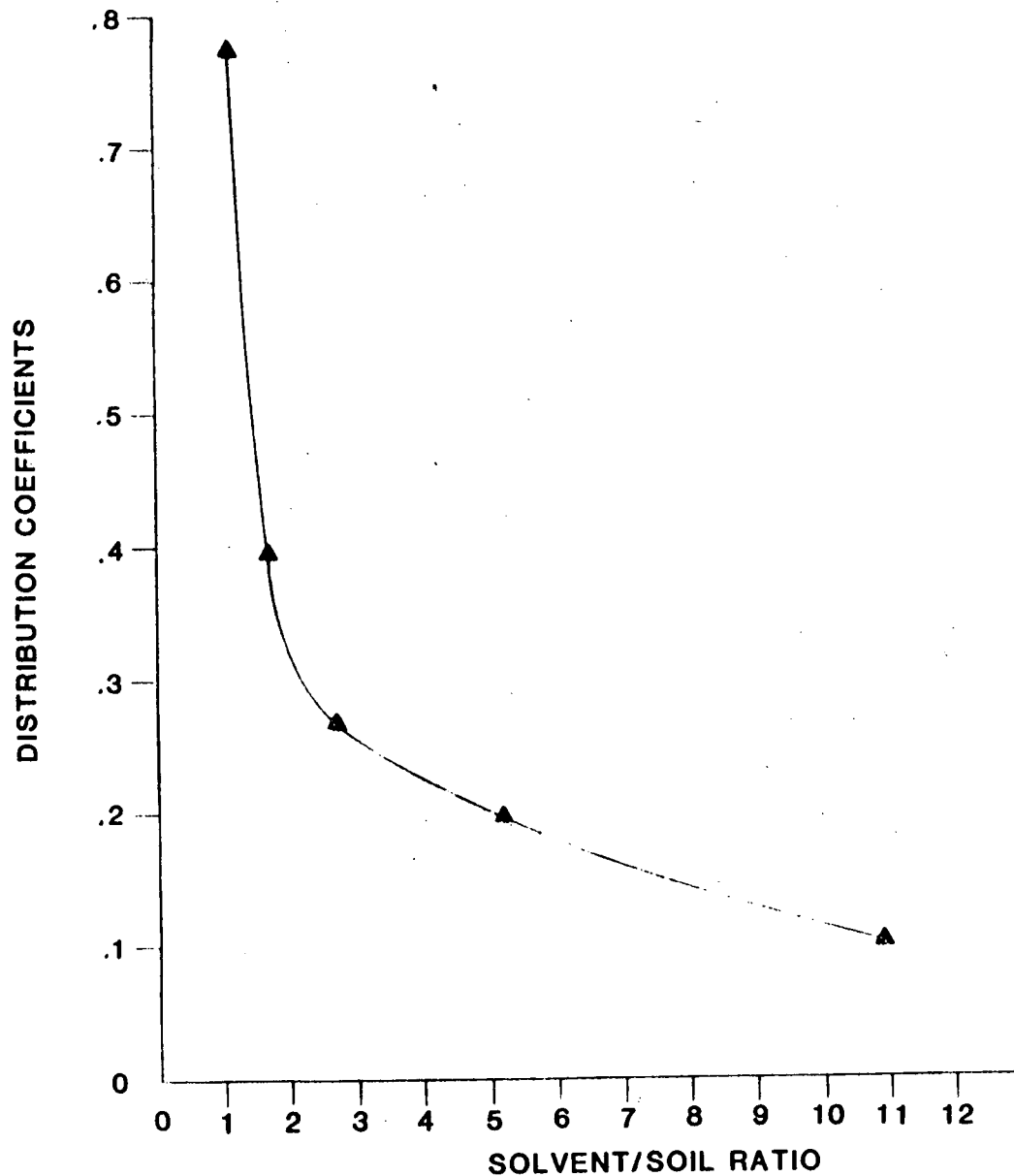
<u>Run</u>	<u>Ratio</u>	<u>PCBs in Solvent</u>	<u>PCBs Remaining In Soil</u>	<u>Percent Reduction</u>	<u>DistCo*</u>
1	1.14:1	235 ppm	80 ug/g	77.1	0.774
2	1.66:1	120 ppm	115 ug/g	67.1	0.395
3	2.76:1	81 ppm	86 ug/g	75.0	0.267
4	5.15:1	60 ppm	33 ug/g	90.6	0.198
5	10.90:1	31 ppm	2.6 ug/g	99.3	0.102

* DistCo is defined as the Distribution Coefficient, a dimensionless value that is the ratio of contaminant solubilized in the solvent to the initial concentration of the contaminant in the substrate.

TABLE 16

EXTRACTION EFFICIENCY OF PCBs VERSUS
SOLVENT/SOIL RATIOS
SOLVENT = 100 PERCENT FREON TF

<u>Run</u>	<u>Ratio</u>	<u>PCBs in Solvent</u>	<u>PCBs Remaining In Soil</u>	<u>Percent Reduction</u>	<u>DistCo</u>
1	2.00:1	66 ppm	225 ug/g	35.7	0.204
2	2.90:1	86 ppm	144 ug/g	58.9	0.266
3	5.00:1	58 ppm	120 ug/g	65.7	0.179
4	9.80:1	37 ppm	100 ug/g	71.4	0.114
5	20.00:1	22 ppm	88 ug/g	74.9	0.068



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**SOLVENT/SOIL RATIO vs.
DISTRIBUTION COEFFICIENTS
100% METHANOL
US-EPA**

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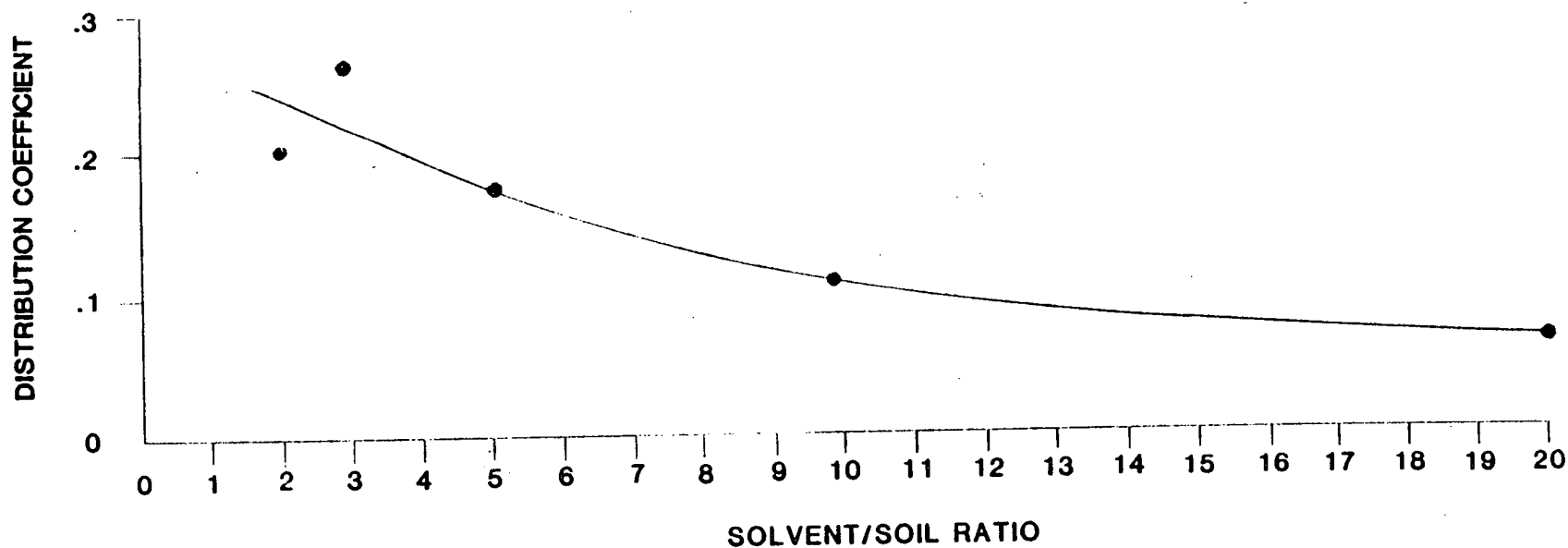
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ORIGINAL
(Red)



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**SOLVENT/SOIL RATIO vs.
DISTRIBUTION COEFFICIENT**

100% FREON

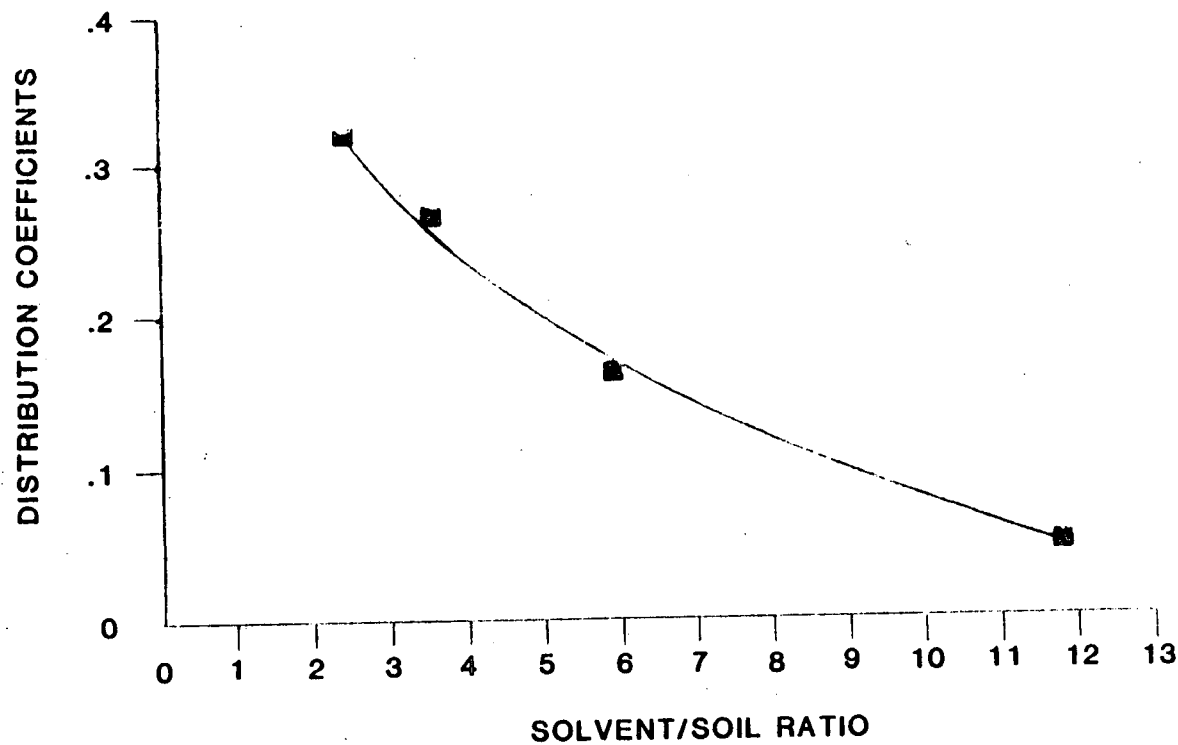
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ORIGINAL (Red)



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**SOLVENT/SOIL RATIO vs.
DISTRIBUTION COEFFICIENTS
50/50 MIXTURE
US-EPA**

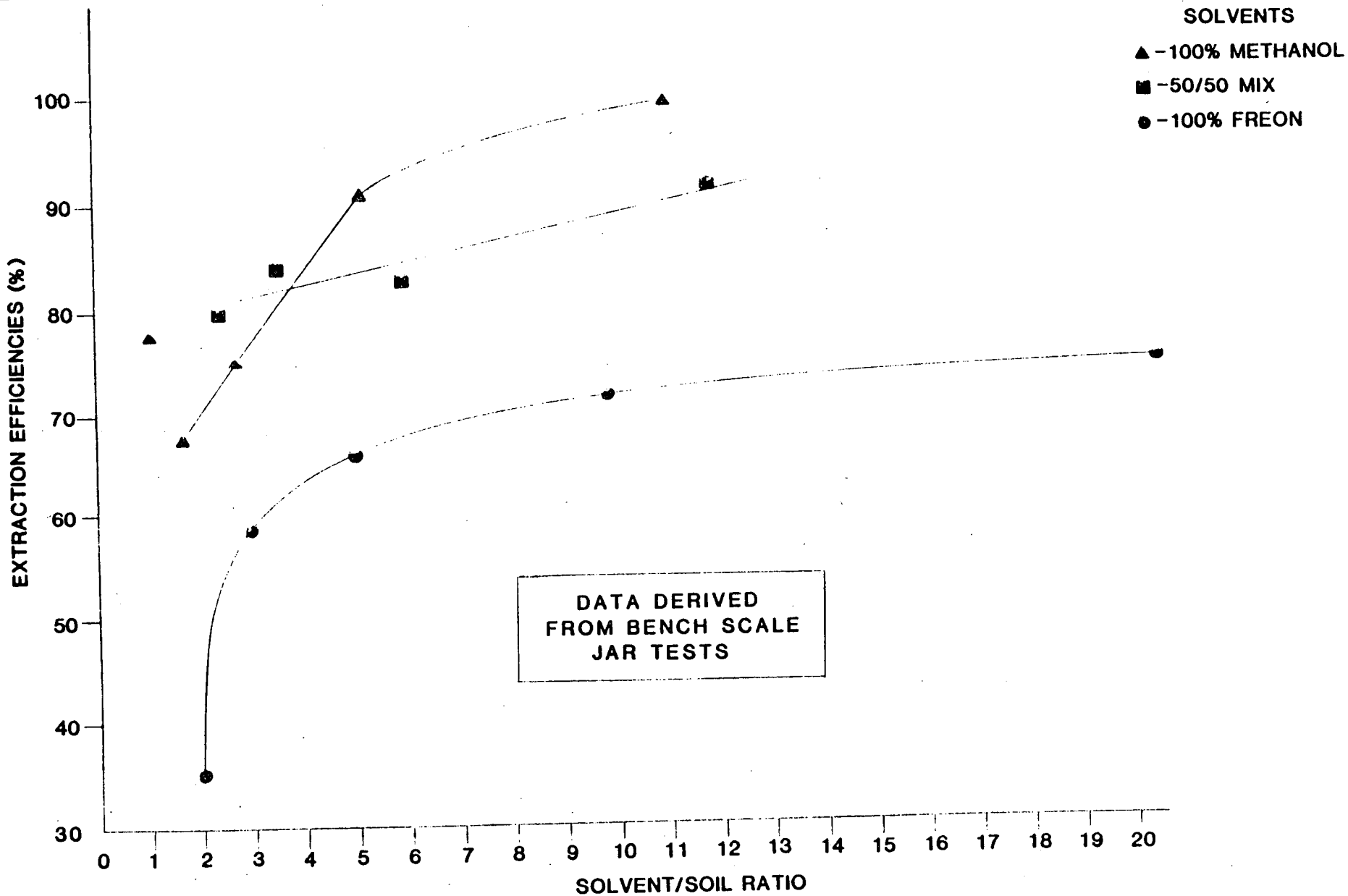
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SOLVENT/SOIL RATIO vs.
EXTRACTION EFFICIENCIES FOR
METHANOL, FREON, 50/50
MIXTURE
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ORIGINAL
(Red)

TABLE 17

EXTRACTION EFFICIENCY OF PCBs VERSUS
SOLVENT/SOIL RATIO
SOLVENT = 50 PERCENT
METHANOL/50 PERCENT FREON TF

Influent PCB concentration = 360 ug/g

<u>Run</u>	<u>Ratio</u>	<u>PCBs in Solvent</u>	<u>PCBs Remaining In Soil</u>	<u>Percent Reduction</u>	<u>DistCo</u>
1	2.40:1	100 ppm	73 ug/g	79.7	0.320
2	3.50:1	83 ppm	58 ug/g	83.9	0.266
3	5.90:1	52 ppm	63 ug/g	82.5	0.166
4	11.80:1	16 ppm	31 ug/g	91.4	0.051

Table 18 gives the results of the benchscale experiment devised to investigate PCB removal as a function of solvent temperature.

TABLE 18

EXTRACTION EFFICIENCY VERSUS TEMPERATURE OF FREON TF

<u>Run</u>	<u>FREON TF Temp. (F)</u>	<u>PCBs Remaining In Soil</u>	<u>PCBs in Solvent</u>	<u>Percent Reduction</u>
1	73 F	89 ug/g	34 ppm	74.6
2	85 F	110 ug/g	31 ppm	68.6
3	100 F	n/a	31 ppm	n/a
4	115 F	100 ug/g	34 ppm	71.4

PCB concentration in the influent soil = 350 ug/g
Initial mass of contaminated soil in each run = 50 grams
Solvent to soil ratio = 10:1

The results of this extraction experiment clearly indicate that raising the temperature of the FREON TF solvent does not improve the extractive efficiency over the range of temperatures tested. This observation is also confirmed by viewing the concentrations of PCBs in the solvent, which are consistently between 31 and 34 ppm. This would suggest that PCB removal efficiencies are relatively constant if all other operational parameters are unchanged.

Pilot Extraction Program (Figure 10)

Tests with 100 Percent FREON TF

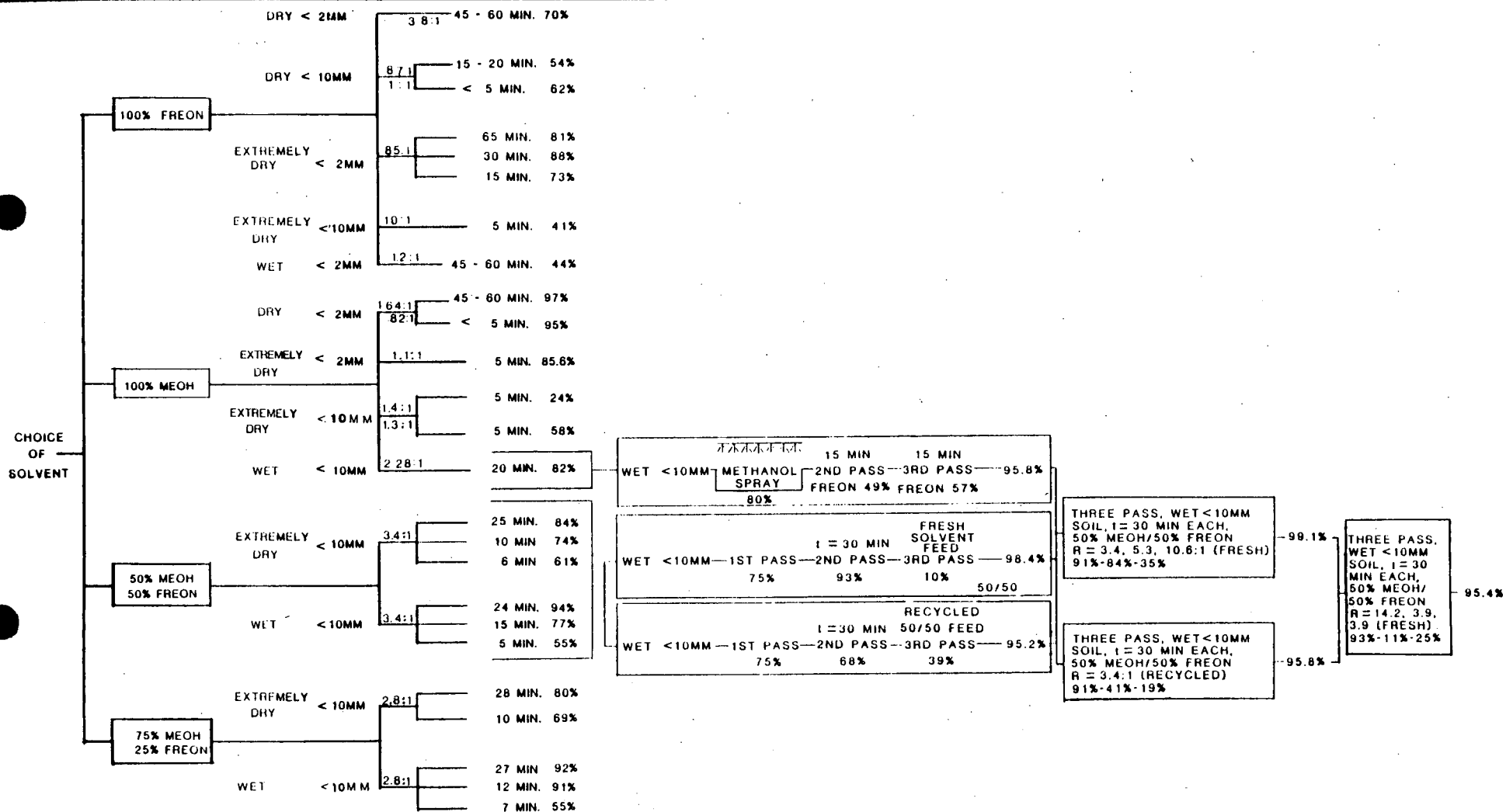
A variety of pilot extraction tests were performed with FREON TF as the solvent. The contaminated soil was prepared so that the entering solvent encountered varying degrees of soil particle size and dryness. The best PCB removal efficiencies occurred for contaminated soil dried extremely well under heat lamps and sieved to contain fractions under 2 mm in size. These reductions were 81 percent, 88 percent, and 73 percent for extremely dry and < 2 mm solids with contactor residence times of 65, 30, and 15 minutes, respectively. The lowest efficiencies were recorded for trials conducted with larger soil particles containing higher degrees of moisture, with reductions as 41 percent, 44 percent, 54 percent, and 62 percent. During the course of tests performed with the FREON TF, influent contaminated soil samples generally averaged between 250 and 400 ug/g of PCBs.

Operators of the contactor equipment noted that the soil particles clumped together upon contact with the FREON TF, regardless of the influent soil moisture content. Trials conducted with the extremely dry contaminated soil, however, did contain a lesser amount of floating particles, except for trials utilizing < 2 mm soil. Overall, the floating particles tended to short-circuit through the pilot contactor and did not indicate a forward progression at all, unlike the settled soil clumps propelled by the stainless steel paddles. Very often, large clumps of FREON TF-wetted soils would cling to the paddles and interfered with the normal forward movement of the heavier-than-FREON TF particles. Residence time in the contactor could only be approximated due to this short-circuiting of particles.

Tests with 100 Percent Methanol

Pilot extraction tests were conducted using methanol as the extractive solvent and the same types of soils prepared for the FREON TF trial runs. With residence times of 45-60, 5, and 5 minutes using dry and < 2 mm soil, removal efficiencies of 97 percent, 95 percent, and 85.6 percent were recorded, respectively. Reductions in PCB levels in the entering soil were lower for trials run with larger particle sizes of soils.

The interaction of the methanol with the contaminated soil was completely opposite to that recorded with FREON TF. Upon mixing, the methanol dispersed the entering soil into fine particles that quickly settled to the bottom of the contactor. Floating particles were not observed throughout the methanol trials. All of these results confirmed results obtained during the benchscale program.



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FLOW SEQUENCE
OF PILOT EXTRACTION TEST

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FIGURE 10

Methanol-wetted soil appeared to move well through the contactor and did not cling to the paddles as did the FREON TF. As a result, the residence time was easily predicted for successive runs. Soil leaving the contactor was moderately wet with methanol and did not dry as fast as soil contacted with the FREON TF. In addition, this solids effluent stream primarily consisted of small coal particles and inerts. Upon inspection following the completion of the methanol trials, the operators observed a layer of fine sediment deposited on the bottom of the GREERCO contactor. These fines apparently were not large enough in size and depth to facilitate a forward progression by the paddles. This was expected since approximately one-eighth of an inch clearance did exist between the paddles and the contactor. The bottom fines were analyzed in later trials for PCB content.

Tests With 50 Percent Methanol/50 Percent FREON TF

Trials were conducted with a mixture of the two solvents in order to take advantage of the methanol/soil dispersion interactions which had been documented during the benchscale studies. These trials utilized contaminated soil that was both wet and dry and less than 10 mm in size.

With the extremely dry and less than 10 mm influent soil, tests showed that relatively high PCB removal efficiencies could be achieved if the contactor residence time approached 30 minutes. This efficiency decreased as the residence time was lowered. Specifically, the reductions observed were 84 percent, 74 percent, and 61 percent for residence times of 25, 10, and 6 minutes respectively. Pilot trials with wet soil demonstrated even greater PCB removal efficiencies. For contactor residence times of 24, 15, and 5 minutes, the observed removal efficiencies were 94 percent, 77 percent, and 55 percent respectively. Solvent to soil ratios were maintained near 3.4:1 during this phase of the investigation, and influent contaminated soil PCB levels averaged between 200 and 300 ug/g. Samples of the effluent solvent stream from the wet soil trials indicated PCB levels from 23 to 35 ppm, while those collected from the extremely dry soil effluent solvent ranged from 44 to 54 ppm.

As with methanol trials, the 50 percent/50 percent mixture used here appeared sufficient to disperse the influent soil into the fine particles as observed in the benchscale studies. Particle buoyancy was not seen and the soils progressed evenly through the contactor. Washed soil leaving the unit dried quickly to yield a powdered residue consisting of soil, coal fines, red dog, and small rocks. Upon the implementation of decontamination procedures for the next series of trials, operators once again observed the layer of fines deposited on the bottom of the contactor trough.

Tests With 75 Percent Methanol/25 Percent FREON TF

Trials conducted with the 75/25 mixture displayed the same visual observations as those recorded for the 50/50 trials. However, PCB removal efficiencies for both wet and extremely dry trials were slightly lower than the values achieved during the 50/50 trials. For extremely dry soil trials, efficiencies recorded were 80 percent and 69 percent at residence times of 28 minutes and 10 minutes, respectively. Wet soil extractive efficiencies totaled 92 percent, 91 percent, and 55 percent for residence times of 27, 12, and 7 minutes, respectively. Solvent to soil ratios were maintained near 2.8:1.

Solvent samples taken in the effluent stream during the wet soil phase showed PCB levels between 37 and 48 ppm, and those for the extremely dry trials were between 46 and 67 ppm. Both sets of figures were significantly higher than the solvent concentrations recorded in similar 50/50 trials; these were likely indicative of the greater methanol percentage utilized.

In the next phase of field pilot experiments, multiple pass trials were performed to investigate the effect of reintroducing previously washed soil for further extractions. The first of these trials entailed a methanol spray wash of the contactor in the contaminated soil, setting a residence time of 5 minutes. The washed soil was collected and, following decontamination of the contactor, contacted with 100 percent FREON TF utilizing a residence time of 15 minutes. The extracted soil was collected again and reintroduced to contact a similar stream of 100 percent FREON TF.

Analytical results demonstrated that the initial methanol spray rinse was successful in extracting 80 percent of the PCBs in the influent soil, with concentration levels decreasing from 430 ug/g down to 85 ug/g. In the next soil pass, the FREON TF further extracted PCBs down to 43 ug/g for an extractive efficiency of 49 percent. The third and final FREON TF pass dropped the concentration of PCBs to 18 ug/g, a 57 percent reduction. Overall, a 95.8 percent reduction in the levels of PCBs was demonstrated. Problems encountered included a tendency of many soil fractions to float in the FREON TF during both passes with this solvent. Significant sticking of soil to the contactor paddles was similarly seen as during the previous trials utilizing 100 percent FREON TF. All three trials were performed with wet and less than 10 mm soil. Residence times for the two FREON TF passes averaged 15 minutes each.

In view of the high PCB extractive efficiencies, poor circuiting, and buoyancy of particles exhibited in the

multiple-pass trials with FREON TF, the next series of tests investigated the reintroduction and subsequent extraction with the 50/50 mixture of solvents.

Each extraction pass was performed with a contactor residence time of approximately 30 minutes and solvent-to-soil ratios of 3.8:1. Results showed that the level of PCBs in the entering wet soil could be lowered from 290 ug/g to 71 ug/g in the first pass, a reduction of 75 percent. A second extraction with fresh solvent brought the PCB concentration down to 5 ug/g in the soil, a 93 percent reduction. The third and final trial of this set only further decreased the level of contamination by 0.5 ug/g down to 4.5 ug/g. The total reduction in PCB levels attained during these three trials was 98.4 percent.

The next series of trials investigated the use of used solvent as the extraction liquid for the second and third passes. Soil introduced to the second extraction stage was the same material used for the second pass described above. During this set of trials, however, the solvent was collected from the previous run and reintroduced with the washed soil. The result was a reduction in PCB concentration from 71 ug/g to 23 ug/g, or 68 percent. The solvent PCB concentration, quantified as 47 ppm after the first pass, increased to 65 ppm following the second. The third pass utilized the 50/50 solvent mixture collected from the second pass. An extractive run performed here with the washed soil saw further decreases in the PCB concentration from 23 ug/g to 14 ug/g, a 39 percent reduction. Samples taken of the spent solvent leaving the third extraction stage yielded a concentration of 66 ppm. Overall, the combination of the three passes with multiple washings and reused solvent demonstrated a removal efficiency of 95.2 percent.

Replicate Trials

The final phase of field pilot extraction studies sought to verify the high removal efficiencies attained in the multiple pass trials in order to provide accurate scale-up data necessary for designing the full-scale treatment system.

The first replicate set of trials was performed using wet contaminated soil which had been screened to contain only the fractions less than 10 mm in size. Fresh 50/50 solvent was introduced for each successive pass at solvent-to-soil ratios ranging from 3 to 10:1. After the first stage, the concentration of PCBs in the soil had been lowered from 500 ug/g to 44 ug/g, a 91.2 percent reduction. This concentration was further lowered to 7.1 ug/g after the second pass, and 4.6 ug/g following the third, for reductions of 84 percent and 35 percent, respectively. Soil fines deposited at the bottom of the contactor contained 7.5 ug/g of PCBs.

A second series of trials was conducted under the same operating conditions except that the solvent was reused for subsequent passes. The first extraction was effective in removing 91.2 percent of the PCBs in the influent soil. Removal efficiencies on the second and third passes were 40.9 percent, and 19.2 percent, respectively. The total combined extractive efficiency was 95.4 percent, and represented a drop in PCB levels from 500 ug/g down to 21 ug/g. PCB levels in the spent 50/50 solvent ranged from 81 to 110 ppm. Residence times were held constant at 30 minutes for all tests.

The final set of pilot extraction trials sought to duplicate the results of earlier multiple pass extractions using fresh 50/50 solvent. Operating parameters were virtually unchanged:

- o Residence time in each stage = 30 minutes
- o Influent contaminated soil wet and screened less than 10 mm
- o Solvent-to-soil ratios relatively unchanged

The results of these trials indicated that this combination of soil extractions was effective in extracting the PCBs from the soil. First-pass efficiency was 93 percent, lowering the concentration from 260 ug/g to 18 ug/g. The next pass lowered the concentration to 16 ug/g, and the third and final pass further reduced this to 12 ug/g. For the combined three-pass system, therefore, the extraction efficiency was 95.4 percent. Samples of the bottom soil fines collected from the bottom of the contactor at the end of the second and third passes showed PCB concentrations of 8.8 ug/g and 13 ug/g, respectively.

IV. DISCUSSION

Initial benchscale studies indicated that the contaminated soils stockpiled in the storage cell consisted of a mixture of fines, coal, red dog, and other materials. These fines were often agglomerated into larger particles due to the presence of oily substances and moisture. When added to FREON TF, this soil initiated buoyancy of large particles at various levels in this non-polar solvent. Methanol, a polar solvent, produced an entirely different effect when added to the contaminated soil. Soil clumps present in the original matrix were broken down and dispersed into fines. These fines settled to yield a layer of sediment at the bottom of the mixture. Buoyant particles were not observed.

Benchscale extraction trials with various ratios of the selected solvents demonstrated that the soil moisture content may indeed affect PCB removal efficiency. For FREON TF as the solvent, removal efficiencies did not exceed 75 percent, while 99+ percent was being realized for the methanol trials over the same range of solvent-to-soil ratios. The differences in extraction efficiency may be due in part to the physical interactions taking place between solvent and soil. FREON TF, a non-polar solvent, repels water and therefore may be slower to extract PCBs from soil containing moisture. In contrast, methanol disperses the soil clumps and probably increases the soil surface area available for extraction, which in turn may help to explain the greater removal efficiencies.

These results were verified during the pilot investigations. Soil which had been previously dried and screened to contain only fractions less than 2 mm was contacted with 100 percent FREON TF, and in a secondary set of trials methanol was substituted. Under these conditions the methanol reductions ranged approximately ten percentage points higher than those recorded with FREON TF. Buoyancy became a problem with the FREON TF as had been recorded in the benchscale experiments. This buoyancy is likely a condition related to the higher density of FREON TF and trapped water in the soil agglomerates. In a continuous, counter-current contactor this buoyancy created short-circuiting of soil through the unit, ultimately leading to lower residence time and non-uniform contact with the solvent. Such an extraction system utilizing 100 percent FREON TF would therefore not be practical in a full-scale capacity.

However, the low boiling point (117 degrees Fahrenheit) of FREON TF and the much higher value for Aroclor 1260 (about 525 degrees Fahrenheit) would make distillation an attractive recycling method in order to reuse the fluorocarbon in repetitive runs.

For this reason, and because of the non-flammable nature of FREON TF, tests continued with this solvent mixed with methanol. Both 50/50 extractions and 75/25 extractions (methanol/FREON) were studied at various residence times and solvent-to-soil ratios. The results show that mixtures containing as low as 50 percent methanol were effective in dispersing the soil agglomerates encountered in the feed stream. Allowing a residence time approaching 30 minutes for both mixtures resulted in extraction efficiencies in the low 90 percentile range. Soil that had been previously dried showed reductions in the middle 80 percentile range, which is consistent with values obtained in the benchscale experiments (Figure 9). These trials have demonstrated that removal efficiencies approaching 90 percent can be achieved for either dry or wet soil conditions encountered at this particular site given the proper residence time. Expected PCB concentrations in the washed soil from a single extraction step would likely range from 50-75 ug/g.

Further pilot testing indicates that multiple extraction passes would be necessary to reduce PCB levels in the soil below this range. These tests were performed while on site and indeed, second and third passes often resulted in residual soil concentrations less than 10 ug/g. Overall removal efficiencies of the first, second, and third passes ranged from 95.2 percent to 99.1 percent. In each set of trials the residence time was set at the value which achieved the optimum results during the earlier experiments, which was 30 minutes.

Test runs were also performed which compared the performance of fresh solvent versus contaminated solvent in multiple-pass systems. These tests show that the 50/50 mixture can be reused several times before its PCB holding capacity is exhausted and the solvent is ready for distillation. Therefore, lower solvent-to-soil ratios can be utilized in the full-scale system as long as the contaminated soil is sufficiently contacted with the liquid.

The full-scale system design will also address the presence of fines in the reused solvent. These are solids which have become entrained in the solvent following contact with the influent soil, and their presence might interfere with subsequent extraction passes. In the full-scale unit a single or series of micron filters could be used to remove these fines. Upon blinding of the filter the collected solids would be returned to the beginning of the treatment system for washing.

Finally, experiments were conducted which investigated PCB removal efficiency as a function of FREON TF solvent temperature. These tests indicated that there were no further reductions as a result of the increased temperatures. However, this is not construed to mean that the same

conclusion would be reached for methanol and the two mixtures utilized. Testing of these and other solvents at higher temperatures was not covered in this study due to time constraints.

All of the data collected from this investigation points toward the use of methanol or a methanol mixture to contact contaminated soil which has been reduced in size for processing. Solvent-to-soil ratios under 5:1 are likely practical with residence times of 30 minutes necessary. The full-scale system must then include provisions for up-front materials conveying, crushing, and screening before extracting with the selected solvent. The solvent feed and recovery system must include provisions for filtering of contaminated fines, recovery of system losses, and recycle of the selected solvent through distillation.

V. DESCRIPTION OF PROPOSED FULL-SCALE TREATMENT SYSTEM

The design of the full-scale treatment system to extract PCBs from the contaminated soil at SHAUFFER reflects the optimum operating conditions achieved during the OHM benchscale and pilot testing performed on site during the latter half of May and early June of 1985. These tests were conclusive in demonstrating that methanol and methanol/FREON TF derivatives are effective in solvents when used in a countercurrent solids/liquid extractor in ratios ranging from 1 to 5 pounds of solvent per pound of influent contaminated soil. The optimum residence time was found to be approximately 30 minutes in the extractor. These and other operating parameters were used in the sizing of a full-scale 10 TPH field extraction system. This proposed full-scale extraction system would remediate PCB soil contamination at SHAUFFER in a time period estimated not to exceed 100 days. The unit process would include provisions for upfront soil classification, conveying, and crushing as the benchscale tests have indicated. The extractor would be sized for 30 minutes of residence time. Specialized explosion-proof pumps would transfer the selected solvent to and from the extraction system. The solvent/soil contacting system would also entail provisions for the containment of volatilized solvents and a vapor condensing unit for recycling purposes. This should minimize drag-out solvent losses and enable makeup volumes to be kept cost-effectively low.

The PCBs solubilized in the liquid could be removed from solution by a variety of techniques including: a) continuous or batch distillation to recover solvent for reuse; and b) granular activated carbon adsorption. PCBs consolidated through distillation of the solvent/PCB mixture could be dealt with in several ways, including a possible on site molten salt destruction unit. Recovered solvent would be recycled for reintroduction into the extraction process with minimal makeup liquid required.

VI. COST ESTIMATE ASSUMPTIONS

Estimate of capital expenditures and operating costs:

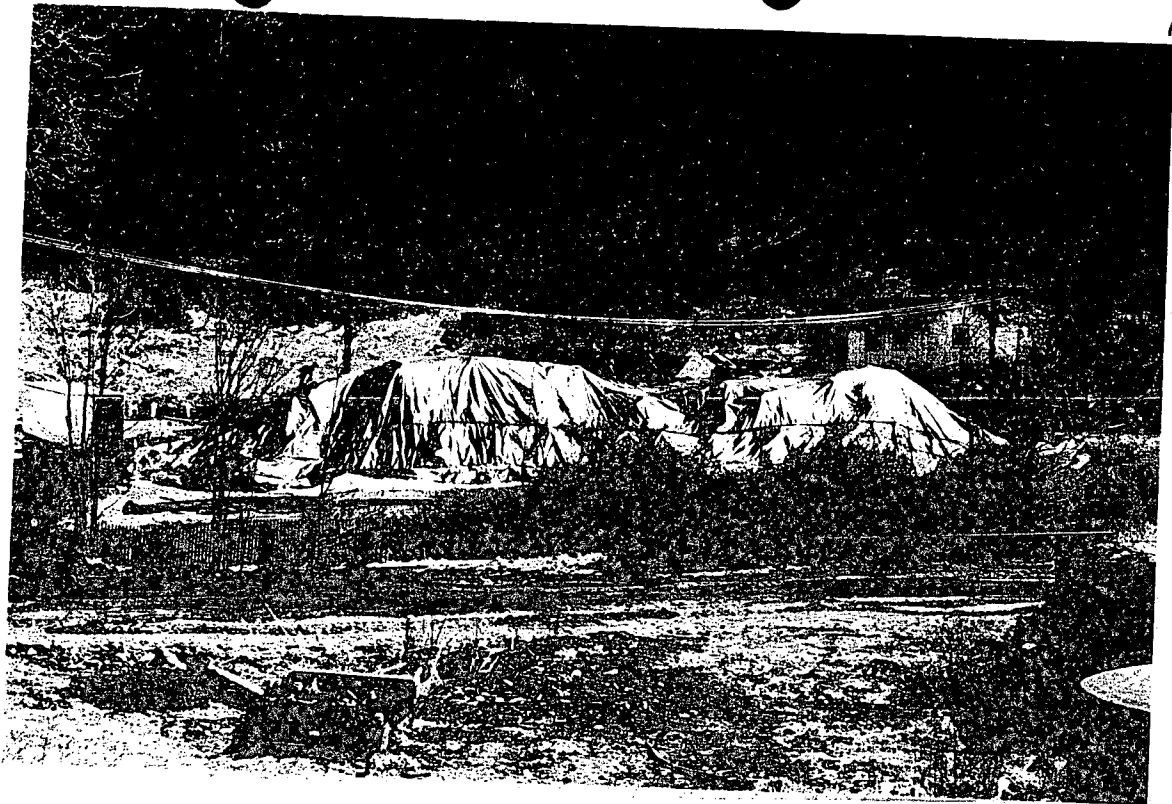
Materials handling equipment	\$	70,000	
Extraction system		130,000	
Chemicals (FREON TF at 2:1, 10% lost)		2,308,500	
(MeOH at 2:1, 10% lost)		<u>192,209</u>	
SUBTOTAL CAPITAL EXPENDITURES WITH FREON USAGE			<u>\$2,508,500</u>
SUBTOTAL CAPITAL EXPENDITURES WITH METHANOL USAGE			<u>\$ 392,209</u>
Personnel	Estimate	\$ 500/hr	
Equipment	Estimate	<u>821/hr</u>	
SUBTOTAL OPERATIONS COSTS			<u>\$ 1,321/hr</u>
Disposal Options			
Molten Salt Destruction Unit:			
Tests not completed to date	\$	n/a	
Carbon Filtration			
121,500 lb at 500 ug/g = 6,075 lb			
PCB at estimated 5% loading on GAC			
= 121,500 lb GAC cost estimate at			
\$1.00/lb		\$ 121,500	
Disposal of Carbon			
Drummed estimate 243 drums at			
\$650/drum		<u>158,000</u>	
SUBTOTAL CARBON			<u>\$ 279,500</u>
Still Bottoms Liquid Incineration			
Assume 5% solvent losses in still			
then 121,500 lb solvent and PCBs			
estimated 383 drums at \$650/drum			
SUBTOTAL BOTTOMS INCINERATION			<u>\$ 249,000</u>

Please note that these costs are inclusive of the line items indicated and may not represent total expenditures associated with these job tasks.

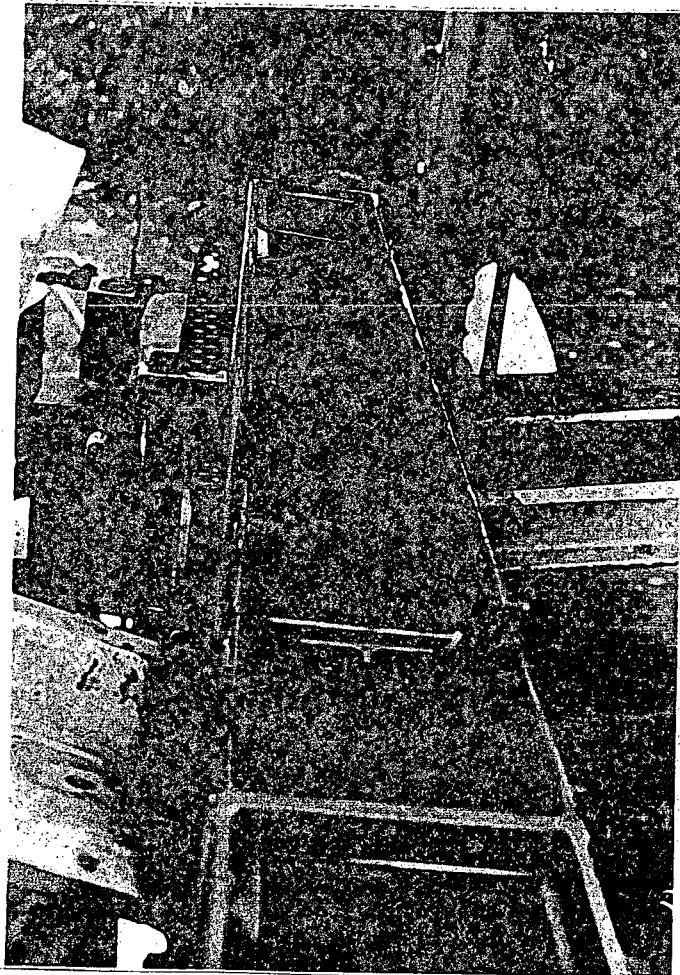
VII. CONCLUSIONS

OHM has proposed a 10 TPH full-scale extraction system to remove PCBs from contaminated soil currently stockpiled at SHAUFFER in Minden, West Virginia. This technique has been investigated during benchscale tests and a pilot treatment program initiated at this site during the latter half of May through early June 1985. The results of this study have clearly indicated that this extraction procedure is technically sound and cost-competitive with conventional means of PCB disposal identified as incineration and land-filling. This new technology is capable of achieving PCB reductions approaching 90 percent for single extractions, with higher efficiencies possible for multiple-pass systems over the range of influent PCB concentrations studied. Several solvents appear to be good candidates for extracting the PCBs, including methanol and methanol/FREON TF mixtures. The selection of an appropriate solvent is, of course, site-specific and may likely prove different for other locations characterized by PCB contamination.

ORIGINAL
(Red)

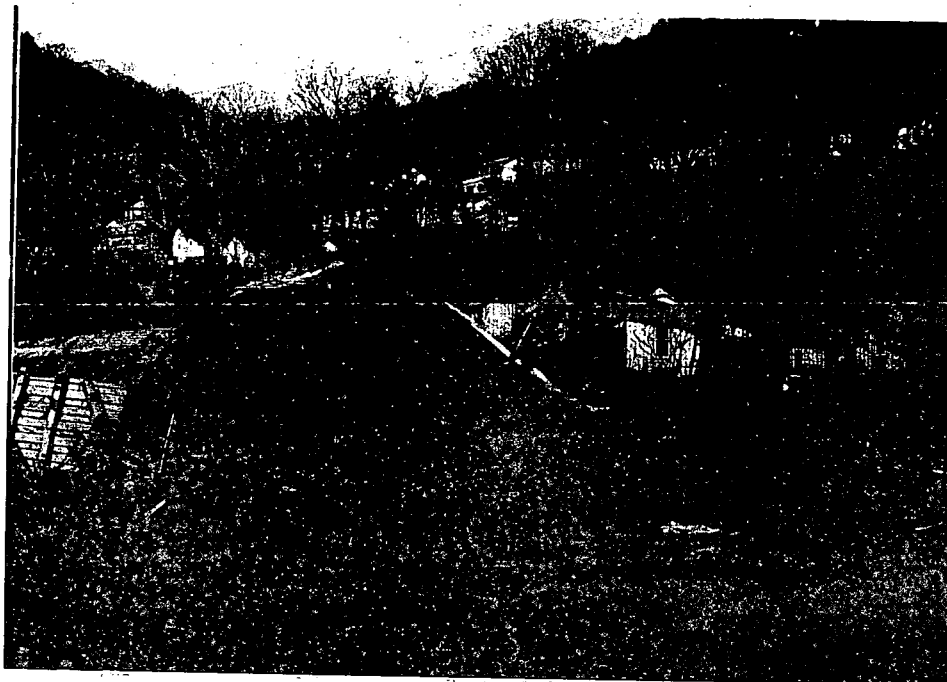


STAGED CONTAMINATED SOIL

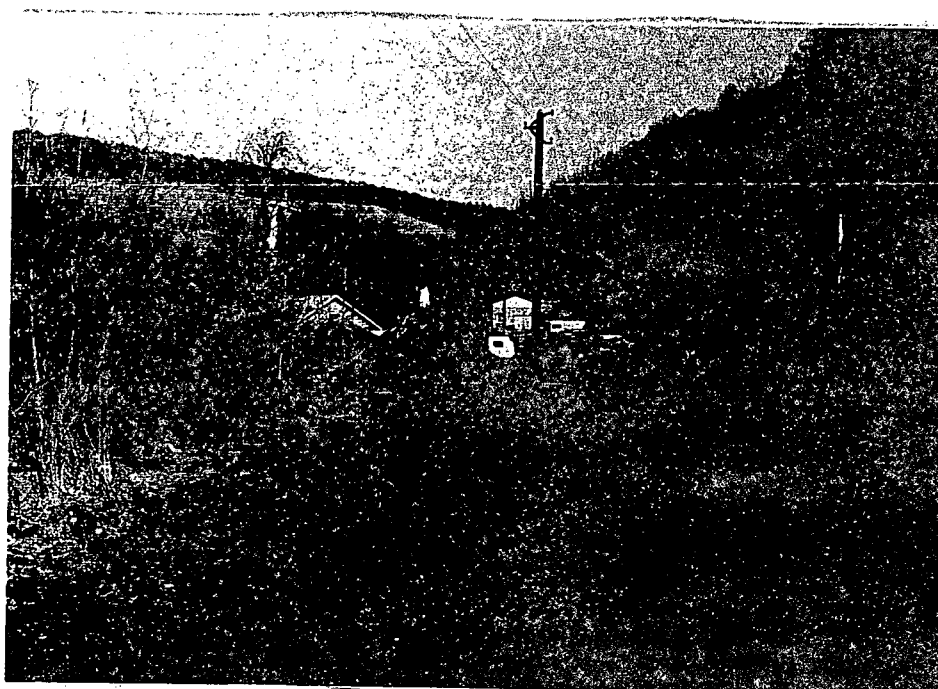


EXAMPLE OF A CONTINUOUS EXTRACTOR

ORIGINAL
(Red)



PROXIMITY OF RESIDENCES



VALLEY LOCATION OF SITE